

Mercury Control with Calcium-Based Sorbents and Oxidizing Agents

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Abstract

Sorbent development work was conducted this quarter in the Catalyst Test Facility at Southern Research Institute, for the purpose of finding optimized compositions of calcium and carbon for mercury removal. Designer sorbents are sought that will allow the sorbent composition to meet the catalytic and sorbent needs of a particular coal type, power-plant configuration, and boiler operation. For example, a utility that produces a high-calcium, low-UBC flyash will need a different composition than a plant that produces a high-UBC, low-calcium flyash. Optimized calcium-based sorbents have been identified and will be tested next quarter for their effectiveness at mercury removal across an ESP, in the Combustion Research Facility (CRF) at Southern Research Institute.

This quarter's results identified the relationship between the degree of mercury removal and the calcium/carbon ratio of calcium-based sorbents. It was found that mercury removal increased dramatically with increasing carbon content up to approximately 10% activated carbon. However, levels of carbon higher than 10% (with balance hydrated lime) resulted in the same amount of mercury removal as those demonstrated for a 10% activated carbon/90% hydrated lime sorbent. Thus, a 10% activated carbon/90% hydrated lime sorbent composition will be a major focus of the pilot-scale CRF tests next quarter. This and other sorbents will be compared with 100% FGD activated carbon. It was also determined from bench-scale tests this quarter that NO and SO₃ concentrations have little impact on the effectiveness of either carbon or calcium-based sorbents for mercury removal. This result for these initial-rate residence-time limited experiments is in contrast to other bench-scale work, where long residence-time, sorbent breakthrough tests were being performed.

The synergistic relationship between carbon and calcium to promote mercury capture by sorbent was explored with several different types of carbon and calcium. Carbon black was found to be highly effective at enhancing mercury removal when mixed with hydrated lime. Activated carbon was somewhat more effective than carbon black. It was also shown that limestone mixed with carbon was as effective as hydrated lime mixed with carbon. Pre-chlorinated calcium-based sorbents were not effective at removing mercury, but mercury removal did increase somewhat with increasing vapor-phase HCl. Results from the benchscale tests this quarter will be used to design a pilot-scale test week in the CRF, where utility flue-gas conditions will exist.

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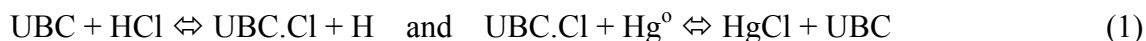
Introduction

The predominant forms of mercury in coal-fired flue gas are elemental (Hg^0) and oxidized (HgCl_2) [1-3]. The percentage of oxidized mercury in the stack effluent of a particular power plant depends on the coal type, combustion efficiency, and the pollution control equipment used. Essentially all of the mercury entering the furnace with the coal is vaporized and exists in the elemental form until the flue gases cool below $\sim 600^\circ\text{C}$ ($\sim 1000^\circ\text{F}$) [1-3]. The oxidation of mercury in coal-fired boiler systems is kinetically limited [1-3]. Where the formation of mercuric compounds is thermodynamically favored, the kinetically controlled oxidation is generally slow unless the oxidant is in vast abundance.

In addition to the trace nature of mercury in coal-fired boilers, favorable reactions for mercury oxidation have short temperature/time windows. Consequently, the extent of mercury oxidation is highly dependent on catalytic processes. Heterogeneous catalysis may enhance mercury oxidation reactions in two ways. First of all, disperse solid catalytic material may provide sorption sites upon which reactions may take place. In addition, heterogeneous catalysis may enhance mercury oxidation by effectively making available gas components (such as Cl) that are otherwise scavenged by competing gas species present at much higher concentrations.

A system of reactions, which include significant chlorine-speciation reactions, has been proposed to describe homogeneous Hg-oxidation [4]. This set of governing reactions allows direct oxidation of Hg^0 to HgCl and HgCl to HgCl_2 by the following four chlorine species with different reaction rates: Cl, Cl_2 , HCl, and HOCl [4]. This system of equations has been shown to effectively predict mercury speciation for specific homogeneous systems [2]. However, the homogeneous model alone consistently under predicts the oxidation of mercury from coal-fired boilers [5]. Nearly all of the chlorine in the flue gas of coal-fired boilers is in the form of HCl [1]. Hydrochloric acid has an extremely low reaction rate with Hg^0 in flue gas. However, catalytic processes can transform some of this HCl into much more reactive forms of chlorine, such as chlorinated carbon sites.

It has been shown in previous work [6,7] that UBC is the dominant component of coal flyash that causes Hg-oxidation enhancement. Niksa et. al. [8] suggested a possible mechanism whereby UBC can catalyze mercury oxidation, as follows:



The March03 and June03 Quarterly Reports show that it is indeed the UBC that is responsible for the observed higher levels of oxidized mercury found in bituminous coal flue gas compared with PRB coal (high-calcium and low-UBC) flue gas [6,9,10].

Considerable evidence has been presented showing that calcium can enhance mercury capture, provided that sufficient UBC is present. Data presented previously suggested that UBC was needed both to catalyze mercury oxidation and enhance mercury capture in the presence of calcium [6,7], and this observation was later firmly established through the means of dual baghouse tests described in the September03 Quarterly Report [11]. It was conclusively shown that there is a synergism between calcium and UBC ($0.1\% < \text{UBC} < 5\%$) that enhances the capture of both oxidized and elemental mercury. Calcium-based sorbents were ineffective at capturing oxidized mercury without carbon present, similar to low-UBC PRB ash [7].

This synergistic relationship between UBC and calcium (either from PRB ash or from injected sorbents) has been observed during coal blending [9-10], but it has been shown to be

much more pronounced for PRB-only tests, where the UBC content was increased via combustion modifications [7, 9-10, 12].

One possible mechanism whereby UBC may enhance mercury capture in the presence of calcium is by providing a sorption site for mercury to attach through van der Waals' forces, thus slowing the mercury molecules down and bringing them in contact or near contact with adjacent calcium sites where reactions may take place. The reaction of elemental mercury with chlorinated carbon sites, i.e., UBC.Cl⁻ (see Equ. 1), followed by reaction with nearby calcium sites, may provide a semi-direct pathway for the capture of elemental mercury by calcium in baghouse filter cakes.

It is true that most (not all) of the UBC present in bituminous coal ash is in separate particles from the rest of the flyash [13], and in a filter cake, the carbon is not intimately associated with the calcium injected as a sorbent. Nevertheless, there may be a sufficient number of contact sites between carbon and calcium, to allow significant mercury capture enhancement to occur. Furthermore, PRB coal ash typically retains its relatively small percentage of UBC on the surface of high-calcium ash particles, thus making the prospects of a two-step reaction (oxidation followed by capture in the case of Hg⁰ and sorption followed by capture in the case of HgCl₂) even more likely. This may be one of the reasons why increasing UBC through combustion modifications while firing PRB coal only was more effective at removing mercury than coal blending. However, the reactivity of PRB UBC is higher than typical bituminous UBC, especially when the PRB UBC is induced by combustion detuning. Hence, carbon type and reactivity may be the major reason why the Hg removal was more effective while firing PRB coal only, than while firing coal blends.

Another theory that has been espoused is that calcium helps the capture of mercury in the presence of carbon by reducing the acid gas. However, if this theory is correct, then a fairly complicated set of mechanisms must be involved, because the enhancement of mercury capture by calcium addition does not correspond directly with the concentration of acid gases (i.e., HCl, SO₂, or SO₃) in the flue gas. In addition, calcium injection has been shown to be ineffective at capturing SO₂ and HCl from flue gas at typical baghouse and ESP temperatures [9].

Most importantly, during bituminous coal firing, UBC has been shown to significantly enhance mercury oxidation, which involves capture of elemental mercury by chlorinated carbon sites and release back into the flue gas, without enhancing mercury capture [7, 9-12, 14]. Hence, the correct theory for explaining the enhancing influence of calcium on mercury capture will describe how the presence of calcium alters the Hg-oxidation and release mechanism (observed for carbon under certain conditions) to a stable-product formation mechanism that produces particulate mercury.

Sorbent development has been pursued this quarter based on these pilot-scale observations of synergism between carbon and calcium in real coal-fired flue gas and under conditions, including temperature/time histories, that are relevant to full-scale boiler systems. The Catalyst Test Facility (CTF) has been used to compare designer calcium-based sorbents with purely-carbon sorbents. Next quarter, optimized sorbents will be tested in the CRF for most effective mercury removal across an ESP.

Experimental

Apparatus

Figures 1 and 2 show pictures of the CTF's tube furnace with quartz micro-reactor, gas-conditioning bubblers for mercury speciation and stabilization prior to mercury monitoring, continuous emission monitors (CEMs), and gas-flow control systems. Both elemental and total mercury are measured at the outlet of the CTF. The CTF simulates clean (no particles) flue gas with all the major flue-gas species present, including CO, CO₂, H₂O, O₂, N₂, HCl, NO, SO₂, SO₃, and Hg⁰, in concentrations that exist in the flue gases of existing power plants, burning specific coal types. The simulated flue gas is originated from compressed gas cylinders and dewars. The gases are mixed to precise concentrations by use of mass flow controllers. Evaporating liquid water generates the appropriate moisture content in the gas stream. Mercury is added to the system using a precisely controlled plenum and reservoir system, where the mercury injection rate is controlled by adjusting the temperature, pressure, and gas flow through the plenum. The simulated flue-gas stream is well mixed and preheated before entering the reaction chamber. A 3"-diameter tube furnace heats the reaction chamber, and a 1½"-tubular reactor carries the gases through the furnace and holds the sorbent samples.

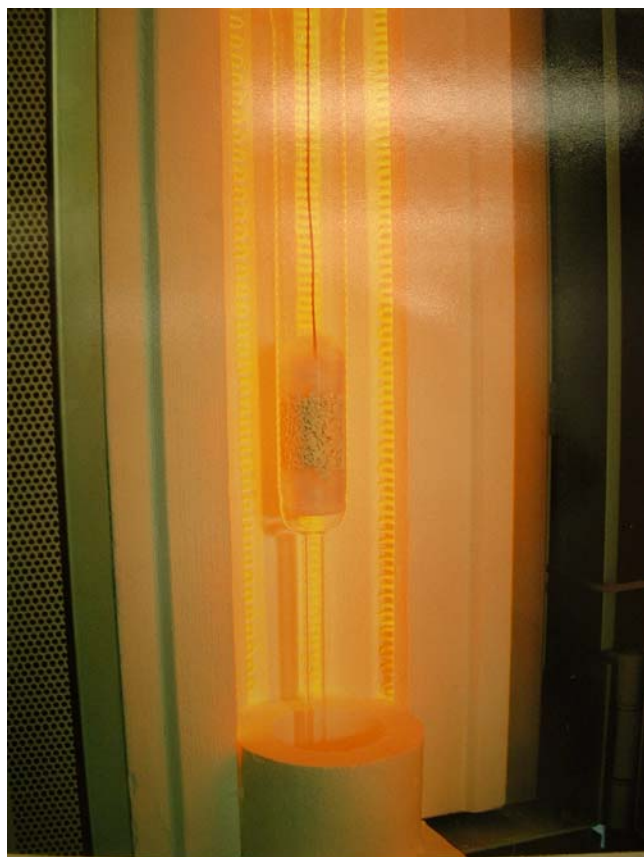


Figure 1. CTF quartz furnace with catalyst at ~1000 °C.

All heated sections of the micro-reactor within the CTF system are made of quartz glass to limit side reactions that might occur as a result of wall effects. A semi-continuous emission

monitor (SCEM) is employed to detect the mercury levels exiting the reaction chamber. A gas-conditioning system is used to convert all Hg into the elemental form, for detection using a combined gold-trap and atomic fluorescence monitor. A Tekran Model 2573A Mercury Vapor Analyzer is used to detect the elemental mercury. Along with mercury, simultaneous measurements of oxygen, carbon dioxide, nitrogen oxides, and sulfur dioxide are made using continuous emission monitors. Figure 3 shows a schematic of the CTF system layout.



Figure 2. CTF furnace, gas-injection system, flue-gas CEMs, and Hg gas-conditioning system.

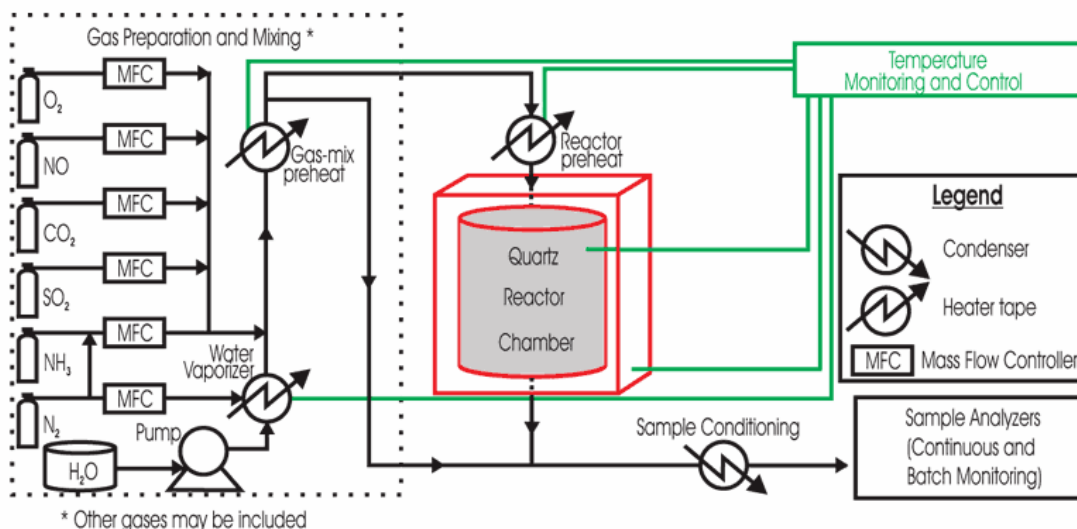


Figure 3. CTF gas flow system.

Even the most rigorous of bench-scale experiments are not sufficient to mimic the full-scale conditions of a coal-fired power plant. Therefore, the CTF was and will continue to be used only for comparison with known effective mercury sorbents and for optimization prior to testing in the pilot-scale unit, where conditions are comparable to full-scale units.

Test method

Residence-time limited experiments were performed in the bench-scale CTF, in order to make kinetic rate comparisons between sorbent types for mercury capture. In addition, it was desired to observe and measure the initial rates of mercury capture by sorbents, before significant acid gases contaminated or otherwise changed the sorbent properties. The objective was to obtain information relevant to sorbent injection and mercury removal across an ESP or a baghouse that is continually being pulsed and refreshed with fresh flyash and sorbent. Under such conditions, the time for acid gases to build up and change the sorbent is limited, and high sorbent utilizations are not obtained. Therefore, a fresh sample in a clean reactor was used for each condition. When a condition was changed, such as temperature, a fresh sample and reactor (identical to that of the previous condition) was quickly installed in place of the previous reactor and sample. In this way, it was possible to obtain mercury capture kinetic information about the sorbents that was somewhat relevant to actual flue-gas conditions. Even so, such bench-scale experiments do have their limitations, and mechanisms suggested at the bench-scale must be tested at the pilot scale, to obtain a complete understanding of the mechanism under full-scale utility flue-gas conditions.

Sorbent utilization and breakthrough experiments were not conducted in this project, primarily because the conditions of such tests are much different than the actual flue-gas conditions for which this project is concerned. Such long residence-time experiments may be more amenable to normalization, to eliminate differences between sorbents, such as: surface area and density. However, such experiments and normalization strip the experimental data of the information desired in this particular project. Furthermore, since the desire here is to obtain information as to how these sorbents will compare in actual utility ESPs and baghouses, the density and surface area of the sorbents were considered inherent properties of the sorbent, and normalization was not desired.

Sorbent types and simulated flue-gas compositions

Table 1 contains a list of sorbents tested this quarter along with a description of the sorbent and its internal surface area. In general, 3-grams of each sorbent were used for each test. However, for particularly effective sorbents, such as activated carbon, some tests were performed with 1.5 or 1.0 grams of sorbent. Surface area was considered to be an inherent property of each sorbent.

Table 2 contains the definition of the three different simulated flue gas conditions examined in this work, for PRB, low-sulfur bituminous, and high-sulfur bituminous coal firing. The bulk gas simulation represents a typical average gas-phase composition of flue gas produced for each coal type.

For specific isolation tests, one of the gas concentrations (i.e., HCl or SO₃) was altered to assess the importance of that particular parameter. In such cases, the condition is reported as the

simulated flue gas that most closely represents that gas composition, and the concentration of the gas component altered is presented expressly.

Table 1. Sorbent Characteristics.

Sorbent Type	Sorbent Description	S. Area (m ² /g)	Ave Part. size (ave)
Activated Carbon (AC)	NORIT Americas Inc. DARCO FGD activated carbon. This is a lignite coal-based activated carbon.	600	90% < 45 μm
Carbon Black (CB)	Continental Carbon 200 Series loose black, Carbon Black. 1.5 μm particles agglomerated from ~30-0.15 μm smoke.	120	1.5 μm
Hydrated Lime	Dravo hydrated lime from Longview Plant in Saginaw.	~18	>10 μm
Limestone (LS)	Dolomitic Limestone from Global Stone Corp.		>10 μm
HL + 20% AC p	Hydrated lime (HL) with 20% AC (processed).		>10 μm
HL + 10% AC p	Hydrated lime with 10% AC (processed).	76	>10 μm
HL + 10% AC m	Hydrated lime with 10% AC (mixed).		>10 μm
HL + 10% CB p	Hydrated lime with 10% CB (processed).		>10 μm
HL + 4% AC p	Hydrated lime with 4% AC (processed).	27	>10 μm
Chl + HL + AC p	Pre-treated hydrated lime with 10% AC with Cl oxidant.		>10 μm
LS + 10% AC m	Limestone (LS) physically mixed with 10% AC.		>10 μm

Table 2. Simulated flue-gas types investigated.

Gas Component	PRB sub-bituminous	Low-S bituminous	High-S bituminous
CO ₂ (%)	15	15	15
CO (ppm)	20	20	20
NO (ppm)	300	300	300
H ₂ O (%)	10	10	10
*O ₂ (%)	0 – 6	0 – 6	0 – 6
SO ₂ (ppm)	500	1000	3000
SO ₃ (ppm)	0 – 30	0 – 30	0 – 30
N ₂ (%)	69 – 75	69 – 75	69 – 75
HCl (ppm)	2	50	100
Hg ($\mu\text{g}/\text{m}^3$)	10	10	10

* Some experiments were conducted without O₂, to avoid oxidation of the sorbent, especially carbon sorbents.

Results

Figure 4 compares the mercury removal capability of two processed calcium-based sorbents, activated carbon/hydrated lime (ACHL) and carbon black/hydrated lime (CBHL). A previous comparison of carbon black and activated carbon showed that carbon black was somewhat less effective as a mercury sorbent than activated carbon [15]. The present test was designed to determine the difference between these two carbon types for use in calcium-based sorbents, where mercury removal is enhanced by a synergistic relationship between carbon and calcium.

The comparison in Fig. 4 suggests that either carbon black or activated carbon could be used to synergistically enhance mercury capture by ash/sorbent mixtures. However, activated carbon appears to be slightly more effective than carbon black.

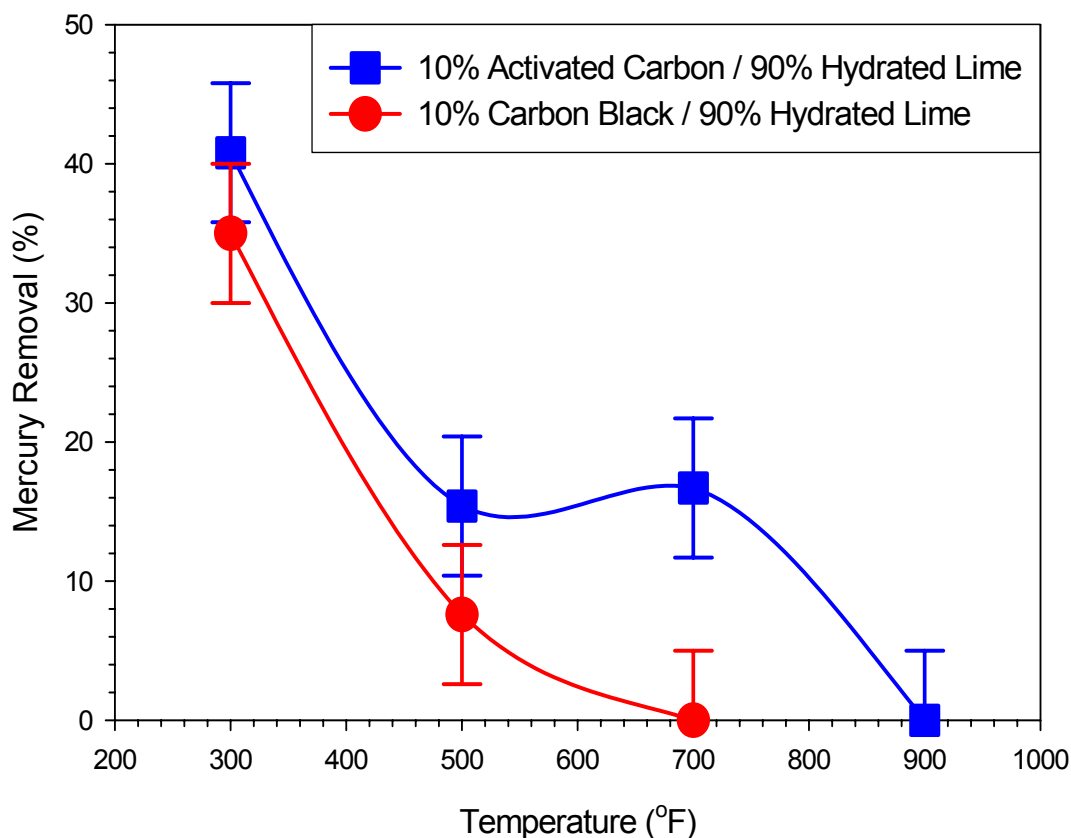


Figure 4. ACHL compared with CBHL.

Several pre-chlorinated sorbents were tested. Figure 5 illustrates the comparison of mercury removal by pre-chlorinated calcium-carbon sorbent with a similar calcium-carbon sorbent that was not pre-chlorinated. The effectiveness of the pre-chlorinated sorbent was tested under several different flue-gas conditions, as shown in Fig. 5. Not only did pre-chlorination of the sorbent not enhance mercury capture, but it hindered mercury capture as well. As illustrated in Fig. 5, the non-chlorinated sorbent performed much better under PRB flue-gas conditions than the pre-chlorinated sorbent, under any flue-gas environment. It is possible that pre-chlorination deactivated the carbon in the sorbent. Pre-chlorinated hydrated lime was also ineffective at removing mercury (see Fig. 5).

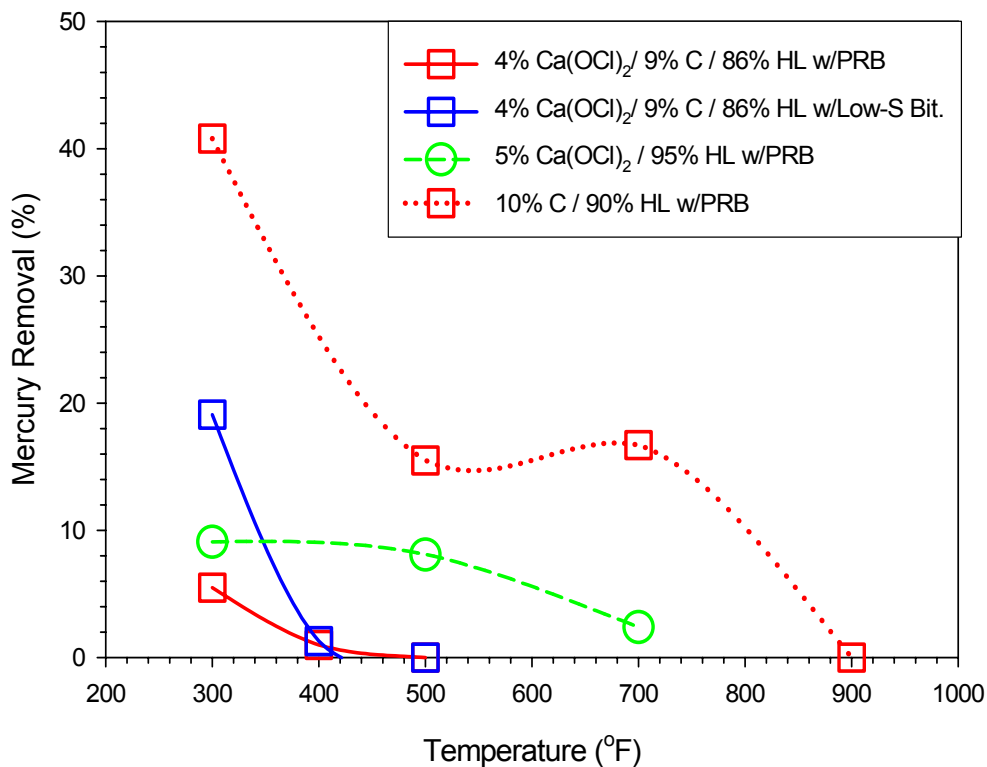


Figure 5. Pre-chlorinated calcium-carbon sorbent compared to non-treated sorbent.

Figures 6 and 7 illustrate that the presence and concentration of NO and/or SO₃ had little, if any, effect on the initial rate of mercury removal by carbon and calcium-based sorbents.

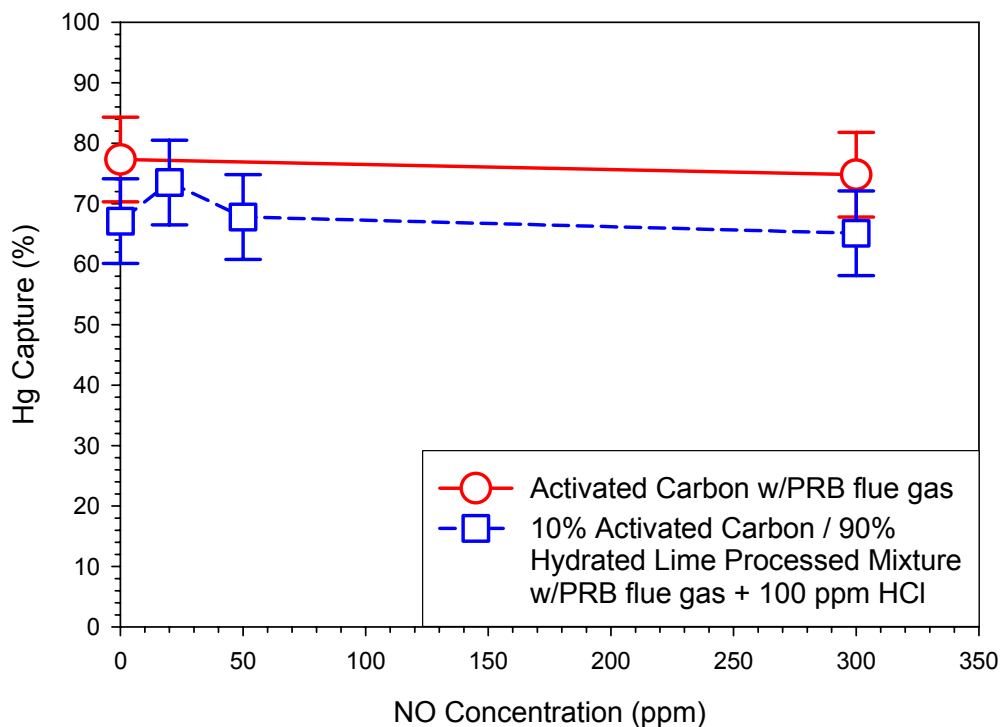


Figure 6. Effect of NO on mercury removal by sorbents.

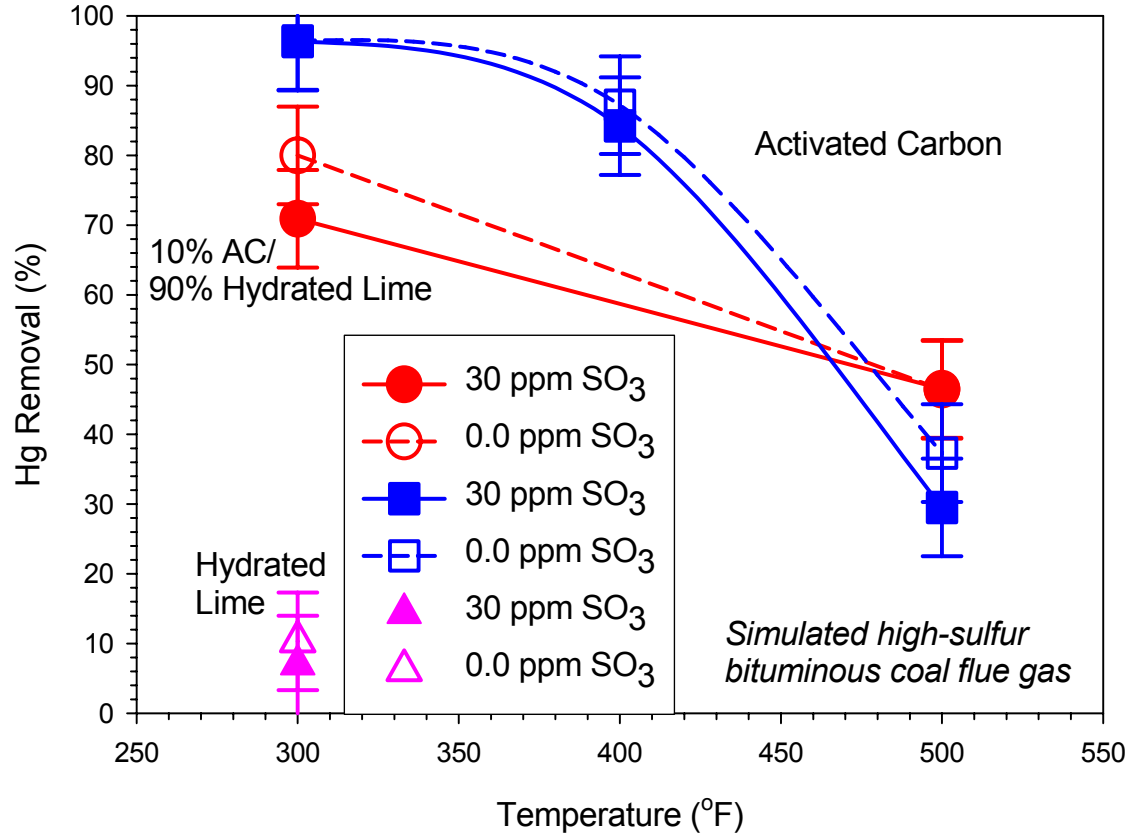


Figure 7. Effect of SO₃ on mercury removal by sorbents.

Such acid gases may have a significant effect on sorbent deactivation or enhancement under long residence-time conditions, where significant time is available for the acid gases to condense and use up the sorbents. However, for practical application of injecting these sorbents in front of an ESP or baghouse, the residence time will be too short, and these gases will not affect the sorbent effectiveness.

Figure 8 illustrates the relationship between C/Ca ratio and mercury capture. For the conditions existing in these short residence-time, bench-scale experiments, mercury removal leveled out at a carbon concentration of 10%. Hence, for the conditions examined, 10% activated carbon with hydrated lime was just as effective as 20% activated carbon with hydrated lime. This phenomenon will be examined further in the pilot-scale CRF test next quarter.

Figure 9 illustrates the relationship between temperature and mercury capture for 10% ACHL under simulated low-S bituminous flue-gas conditions. Each effective sorbent investigated in this work was much more effective at removing mercury at the lowest temperature condition, 300 °F, than at higher temperatures. The effectiveness of the ACHL sorbent illustrated in Fig. 9 ends at ~500 °F. However, for different flow rates and gas compositions, this and other sorbents retained some effectiveness at still higher temperatures (see Fig. 7).

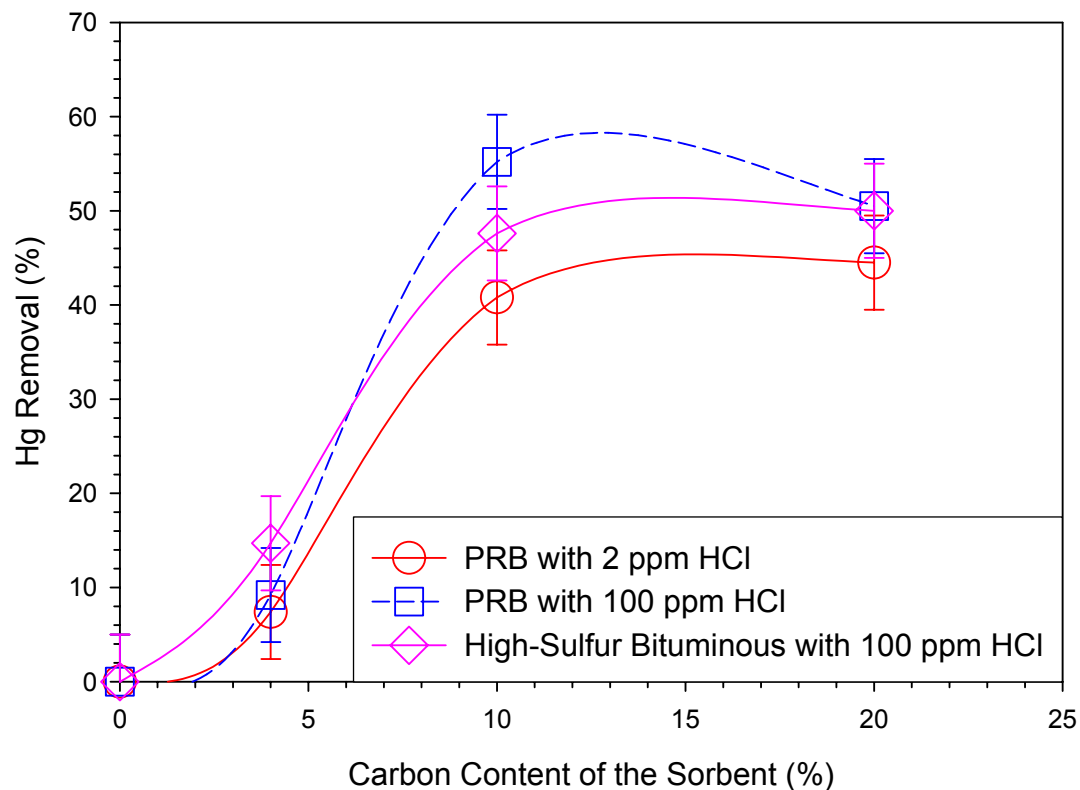


Figure 8. Mercury removal versus carbon content of calcium-based sorbents.

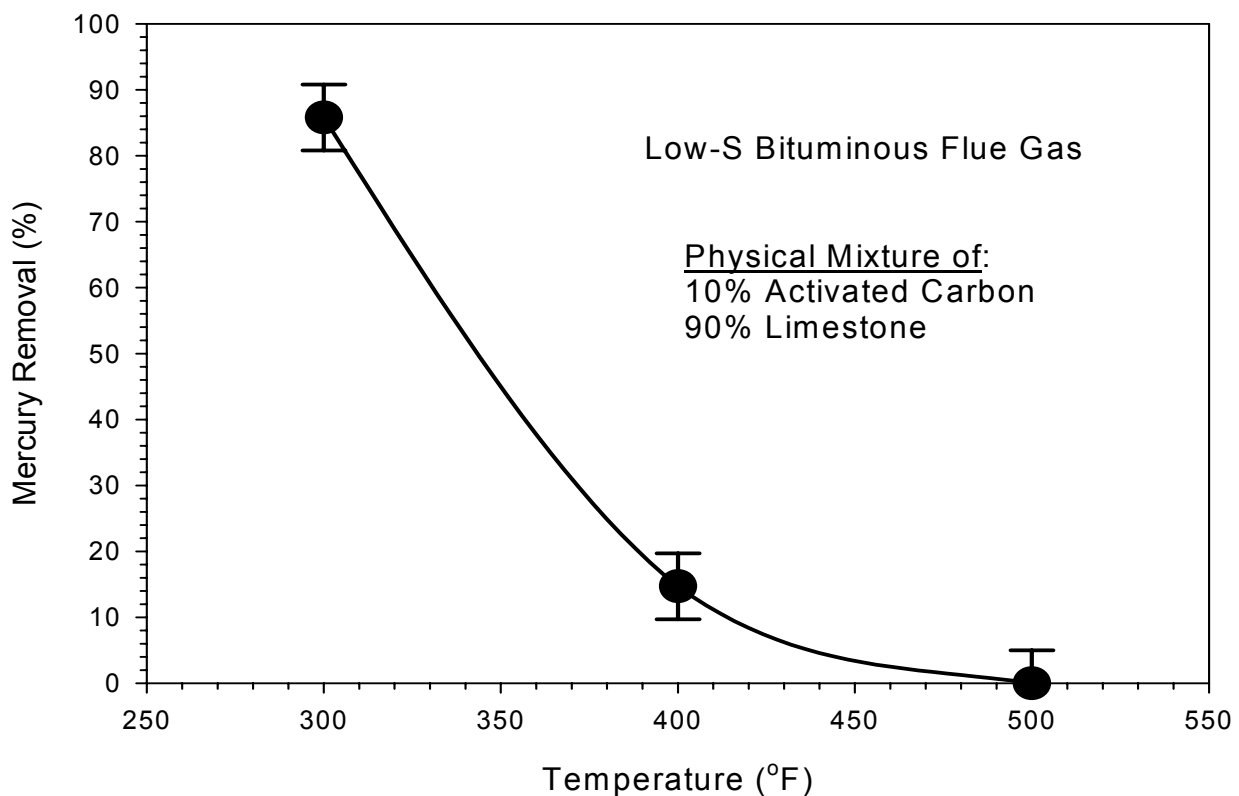


Figure 9. Temperature dependence of Hg-capture by ACLS sorbent.

Figure 10 illustrates the results for mercury capture by six different sorbents. Limestone, consistent with the results for hydrated lime, was not an effective mercury sorbent by itself. However, as was hydrated lime, limestone mixed with a small amount of activated carbon was an effective sorbent. In fact, a mixture of limestone and activated carbon was more effective at removing mercury than a mixture of hydrated lime and activated carbon, compared on an equal mass basis. It is presumed that limestone was more effective because it has a higher density than hydrated lime and would create a fixed bed such that the simulated flue gas was forced to experience more intimate contact with the carbon than for the hydrated lime/carbon sorbent. Nevertheless, PRB ash, hydrated lime, and limestone have all been shown to be effective at enhancing mercury capture when carbon is present [6-7, 9-12, 14]. Limestone/carbon sorbents will be tested next quarter in the pilot-scale facility.

Figure 10 also illustrates that physical mixtures of calcium and carbon were just as effective as processed mixtures. In fact unmixed layering of calcium on carbon or carbon on calcium obtained essentially the same mercury removals as well-mixed sorbents and processed sorbents. These results suggest that calcium did not participate in the mercury capture process directly, in the CTF bench-scale experiments. On the other hand, pilot-scale tests have conclusively shown that calcium can enhance the capture of both elemental and oxidized mercury when carbon is present [12]. It is not possible to completely mimic full-scale conditions in bench-scale apparatus, and the governing mechanisms are somewhat different between the bench-scale apparatus and the full- or pilot-scale experiments.

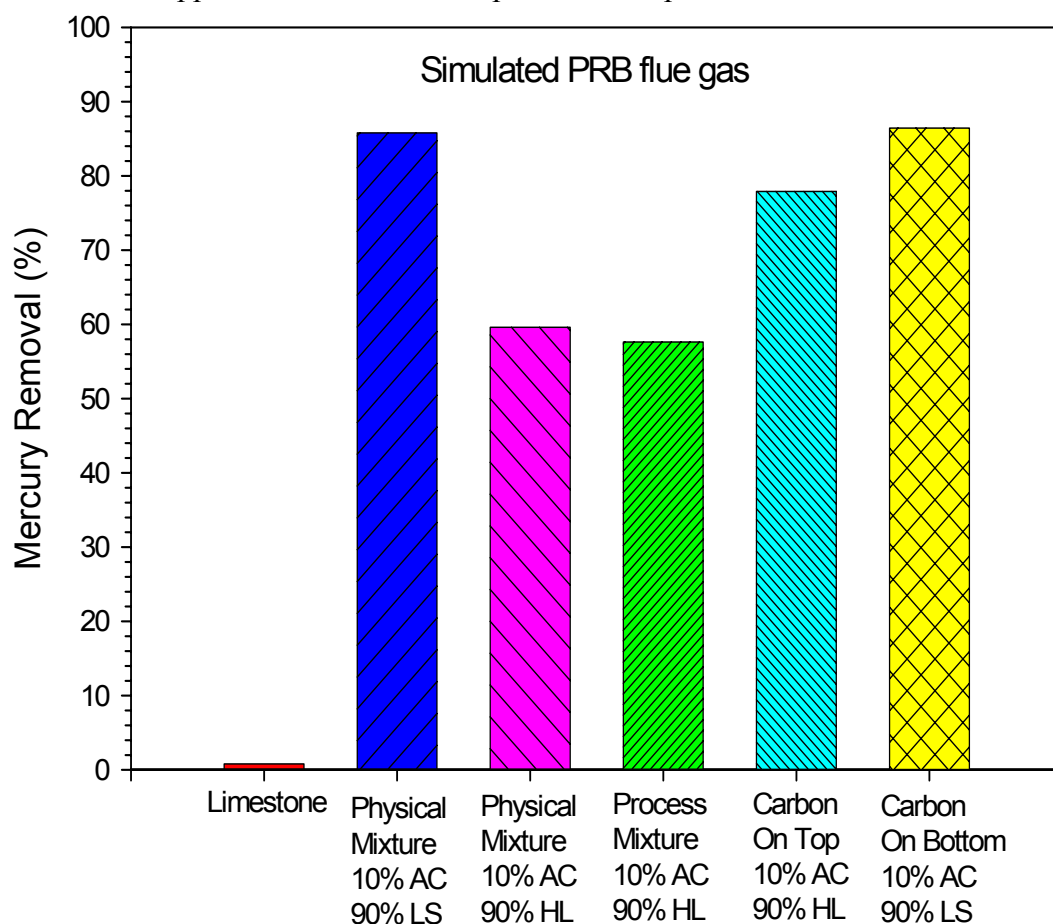


Figure 10. Limestone and physical mixtures of carbon and calcium compared.

It may be that the synergistic relationship between carbon and calcium, observed in the pilot-scale experiments, is suppressed or less dominant in the small-scale CTF. Fundamental investigation of Ca/C synergism mechanisms for Hg capture will resume at the pilot-scale next quarter.

Conclusions

The bench-scale sorbent-comparison experiments this quarter identified a Ca/C sorbent composition range for optimum mercury removal. It was found that 10% activated carbon/ 90% hydrated lime (10% ACHL) was much more effective at removing mercury than 4% activated carbon/ 96% hydrated lime. However, a 20% activated carbon / 80% hydrated lime (20% ACHL) sorbent had about the same effectiveness for mercury removal as the 10% ACHL. Hence, pilot-scale tests during the next quarter will examine the effectiveness of 10% ACHL at removing mercury across an ESP. The 20% ACHL sorbent will also be investigated, given that the governing mechanisms in the actual flue gas are different than in the bench-scale facility.

It was also discovered that limestone is as effective an admixture as hydrated lime for enhancing mercury removal on Ca/C sorbents, in the bench-scale facility. Limestone/carbon sorbent (i.e., 10% LSAC) will also be tested in the pilot-facility next quarter. It was also shown, consistent with previous pilot-scale tests, that NO and SO₃ had no effect on the ability of carbon or calcium-based sorbents to capture mercury at temperatures of 300 °F and upward. Finally, pre-chlorinated sorbents were found to be ineffective at removing mercury.

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